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PATENT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re application of:

Zhenhua MAO, et al

Serial No.:

10/501,311

Group No.:

Filed:

July 13, 2004

Examiner:

For:

COATED CARBONACEOUS PARTICLES PARTICULARLY USEFUL AS ELECTRODE MATERIALS IN ELECTRICAL STORAGE CELLS AND METHODS OF MAKING THE SAME

Attorney Docket No.;

U 015296-2

Commissioner for Patents

P. O. Box 1450

Alexandria, VA 22313-1450

REQUEST FOR REFUND

Deposit Account 12-0425 was charged \$290.00 for multiple dependent claims (Fee Code 1616) and \$1,062.00 for fifty-nine (59) extra claims over twenty (Fee Code 1615) on June 3, 2005 (Control Nos. 22 & 24).

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However, no claim fee charge was authorized and according to 37 CFR 12492(g), if additional fees are required they only must be paid prior to the expiration of the term period set for reply by the Office in any Notice of Fee deficiency, which has not issued.

In the meantime, a Preliminary Amendment cancelling the multiple dependent claims (copy attached) was filed on Completion of the Filing Requirements on June 21, 2005. This application has a total of fifty-two (52) extra total claims. A copy of the acknowledgment postcard stamped by the Patent Office is attached.

Therefore, refund of the total charges of \$416.00 by credit to Deposit Account 12-0425 is requested.

Respectfully submitted,

JOHN RICHARDS

C/O LADAS AND PARRY LLP

26 WEST 61ST STREET NEW YORK, N Y 10023

REG. NO. 31,053 (212) 708-1915

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I hereby certify that this Completion of Filing Requirements and the papers indicated as being transmitted therewith are being deposited with the United States Postal Service on this date <u>June 21, 2005</u>, in an envelope as "Express Mail Post Office to Addressee," Mailing Label Number <u>EV480460256US</u>, addressed to the: Commissioner for Patents, P. O. Box 1450, Alexandria,

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IN THE CLAIMS:

- 1. (Original) A process for the production of coated carbonaceous particles comprising: providing a carbon residue forming material selected from materials which, when oxidized and then thermally decomposed in an inert atmosphere to a carbonization temperature of at least 850° C, form a residue which is at least 90 wt. % carbon; providing particles of a carbonaceous material; mixing the carbon residue forming material and the particles of the carbonaceous material under conditions sufficient to deposit a uniform coating of the carbon residue forming material onto the surface of the carbonaceous particles; and stabilizing the coated particles by subjecting the coated particles to an oxidation reaction.
- 2. (Original) A process for the production of coated carbonaceous particles comprising: providing a first solution of a carbon residue forming material, wherein the first solution comprises one or more solvents and a carbon residue forming material selected from materials which, when oxidized and then thermally decomposed in an inert atmosphere to a carbonization temperature of at least 850 °C, form a residue which is at least 90 wt.% carbon; providing particles of a carbonaceous material; mixing the first solution of the carbon residue forming material and the particles of the carbonaceous material at an elevated temperature; depositing a coating of the carbon residue forming material onto the surface of the carbonaceous particles; and stabilizing the coated particles by subjecting the coated particles to an oxidation reaction.
- 3. (Original) The process of claim 2, wherein the particles of a carbonaceous material are supplied as a dispersion in a second solution comprising one or more solvents before mixing with the first solution.
- 4. (Original) The process of claim 2, further comprising adding one or more solvents to the mixture of the first solution and the particles.
- 5. (Currently Amended) The process of claim[[s]] 1 or 2, further comprising carbonizing the coated particles.

- 6. (Original) The process of claim 5, wherein the coated particles are carbonized in an inert atmosphere at a temperature of greater than about 400 °C.
- 7. (Original) The process of claim 5, wherein the coated particles are carbonized in an inert atmosphere at a temperature of between about 400° C to about 2000 °C.
- 8. (Original) The process of claim 5, further comprising graphitizing the coated particles.
- 9. (Currently Amended) The process of claim[[s]] 1 or 2, further comprising graphitizing the coated particles.
- 10. (Original) The process of claim 9, wherein the coated particles are graphitized at a temperature of greater than about 2000° C.
- 11. (Original) The process of claim 9, wherein the coated particles are graphitized at a temperature of greater than about 2000° C.
- 12. (Original) The process of claim 2, wherein the coated particles are separated from the mixture of the first solution of the carbon residue forming material and the particles of a carbonaceous material.
- 13. (Original) The process of claim 12, wherein the coated particles are washed and dried.
- 14. (Original) The process of claim 12, further comprising recovering the one or more solvents from the mixture.
- 15. (Original) The process of claim 14, wherein the one or more solvents are recovered by one or more steps selected from the group consisting of distillation or evaporation.

- 16. (Original) The process of claim 14, wherein the recovered solvent is reused in the first solution of carbon residue forming material.
- 17. (Original) The process of claim 2, wherein the solvent is selected from the group consisting of toluene, benzene, xylene, quinoline, tetrahydrofuran, tetrahydronaphthalene, naphthalene, methanol, acetone, 1-methyl-pyrrolidinone and water.
- 18. (Original) The process of claim 2, wherein the first solution is mixed at an elevated temperature to dissolve the carbon residue forming material in the one or more solvents.
- 19. (Original) The process of claim 3, wherein the second solution is mixed at an elevated temperature.
- 20. (Original) The process of claim 2, wherein the ratio of the one or more solvents to the carbon residue forming material in the first solution is 1: 1 or less.
- 21. (Original) The process of claim 3, wherein the ratio of the one or more solvents to the carbon residue forming material in the mixture of the first and second solutions greater than 2:1.
- 22. (Original) The process of claim 3, wherein the ratio of the one or more solvents to the carbon residue forming material in the mixture of the first and second solutions is greater than about 5: 1.
- 23. (Original) The process of claim 1, wherein the coating of carbon residue forming material is deposited under ambient or higher pressure.
- 24. (Original) The process of claim 23, wherein the coating of carbon residue forming material is deposited at a temperature of about -5°C to about 400 °C.

- 25. (Original) The process of claim 1, wherein the carbonaceous particles comprise a pulvurent carbonaceous material selected from the group consisting of petroleum pitches, calcined petroleum cokes, uncalcined petroleum cokes, highly crystalline cokes, coal tar cokes, synthetic graphites, natural graphites, soft carbons derived from organic polymers, and soft carbons derived from natural polymers.
- 26. (Original) The process of claim 1, wherein the particles have an average particle size of up to about $150\mu m$.
- 27. (Original) The process of claim I, wherein the particles have an average particle size of between about $5\mu m$ to about $70 \mu m$.
- 28. (Original) The process of claim1, wherein the particles have an average particle size of between about $5\mu m$ and about $45\mu m$.
- 29. (Original) The process of claim 2, wherein the carbon residue forming material is deposited onto the surface of the particles by selectively precipitating the carbon residue forming material onto the particles.
- 30. (Original) The process of claim1, wherein the carbon residue forming material is a polymeric material selected from the group consisting of heavy aromatic residues from petroleum and coal of chemical processes, lignin from pulp industry, phenolic resins, and carbohydrate materials.
- 31. (Original) The process of claim 1, wherein the carbon residue forming material is selected from the group consisting of petroleum pitches and coal tar pitches or pitches produced by chemical processes.
- 32. (Original) The process of claim 1, wherein the carbon residue forming material is deposited onto the surface of the particles in amounts of between about 1% and about 50% by weight.

- 33. (Original) The process of claim 1, wherein the carbon residue forming material is deposited onto the surface of the particles in amounts of between about 2.5% and 25% by weight.
- 34.(Original) The process of claim 1, wherein the oxidation reaction is carried out in the presence of an oxidizing agent.
- 35. (Original) The process of claim 34, wherein the oxidizing agent is a solid oxidizing agent selected from the group consisting of inorganic and organic oxidizing agents.
- 36. (Original) The process of claim 35, wherein the solid oxidizing agent is selected from the group consisting of alkali nitrates and alkali sulfates.
- 37. (Original) The process of claim 37, wherein the solid oxidizing agent comprises a compound having the formulaM'OX, wherein M' is selected from the group consisting of transition metals, inorganic salts, and organic salts, and wherein x is an integer between _0.5_ and _3_.
- 38. (Original) The process of claim 34, wherein the oxidizing agent is a liquid oxidizing agent.
- 39. (Original) The process of claim 38, wherein the liquid oxidizing agent is selected from the group consisting of oxidizing acids, aqueous solutions containing oxidizing salts, non-aqueous solutions containing oxidizing salts, peroxides and aryl quinones.
- 40. (Original) The process of claim 34, wherein the oxidizing agent is a gaseous oxidizing agent selected from the group consisting of oxygen, sulfur fumes, gaseous oxides, nitrogen oxide gas, ambient air, and halogens.
- 41. (Original) The process of claim 34, wherein the oxidation is carried out at elevated temperatures.
- 42. (Original) The process of claim 41, wherein the elevated temperature is supplied in a controlled

manner with temperature ramps and hold periods.

- 43. (Original) The process of claim 34, wherein the oxidation is carried out under reduced pressure.
- 44. (Original) Coated carbonaceous particles comprising a coating layer formed of an oxidized fusible carbon residue forming material.
- 45. (Original) The coated carbonaceous particles of claim 44, wherein the coating layer is formed of a graphitized, fusible oxidized carbon residue forming material.
- 46. (Original) The coated carbonaceous particles of claim 44, wherein the particles comprise a substantially smooth coating.
- 47. (Original) The coated carbonaceous particles according to claim 44, wherein the carbonaceous particles comprise a pulvurent carbonaceous material selected from the group consisting of petroleum pitches, calcined petroleum cokes, uncalcined petroleum cokes, highly crystalline cokes, coal tar cokes, synthetic graphites, natural graphites, soft carbons derived from organic polymers, and soft carbons derived from natural polymers.
- 48. (Original) The coated carbonaceous particles according to claim 44, wherein the carbonaceous particles are a pulvurent carbonaceous material selected from the group consisting of calcined petroleum cokes, uncalcined petroleum cokes, highly crystalline cokes, synthetic graphites, and natural graphites.
- 49. (Original) The coated carbonaceous particles of claim 44 wherein the carbonaceous particle and the coating layer are both graphitic.
- 50. (Original) A method for the production of a Li-ion battery wherein the coated carbonaceous particles of claim 44 are used as the anode material, and wherein such Li-ion battery exhibits a first

cycle charge efficiency greater than 90% at the cut-off potential of 1 volt versus Li when tested with electrolyte containing no propylene carbonate solvent.

- 51. (Original) An electrical storage cell comprising the coated carbonaceous particles of claim 44.
- 52. (Original) An electrical storage cell according to claim51, wherein the electrical storage cell is a rechargeable electrical storage cell.
- 53. (Original) A method for the manufacture of an electrical storage cell which comprises incorporating the coated carbonaceous particles of claim 44 into an anode of the electrical storage cell.
- 54. (Original) A process for the production of coated carbonaceous particles having substantially smooth coatings formed of an oxidized, carbon residue forming material comprising: providing a first solution of a carbon residue forming material selected from the group consisting of petroleum pitches and coal tar pitches, wherein the first solution comprises one or more solvents selected from the group consisting of toluene, xylene, quinoline, tetrahydrofuran, tetrahydronaphthalene, and naphthalene; providing particles of a carbonaceous material selected from the group consisting of calcined or un-calcined petroleum cokes, natural graphite and synthetic graphite, wherein the particles are provided in a second solution comprising one or more solvents; mixing the first solution and the second solution at an elevated temperature; depositing a coating of the carbon residue forming material onto the surface of the particles; and stabilizing the coated particles by subjecting the particles to an oxidation reaction; and carbonizing the coated particles.
- 55. (Original) The process of claim 54 in which the carbonaceous material is selected from calcined or uncalcined petroleum cokes, further comprising graphitizing the coated particles.
- 56. (Original) The process of claim 54, wherein the coated particles are carbonized in an inert atmosphere at a temperature of greater than about 400° C.

- 57. (Original) The process of claim 54, wherein the coated particles are carbonized in an inert atmosphere at a temperature of between about 550°C to about 1500°C.
- 58. (Original) The process of claim 55, wherein the coated particles are graphitized in an inert atmosphere at a temperature of greater than about 2500° C.
- 59. (Original) Coated carbonaceous particles comprising a coating layer formed of an oxidized, carbon residue forming material produced by the process of claim 54.
- 60. (Original) Coated graphitic particles comprising a coating layer formed of an oxidized, carbon residue forming material produced by the process of claim 55.
- 61. (Original) An electrical storage cell comprising the coated graphitic particles of claim 60.
- 62. (Original) An electrical storage cell according to claim 61, wherein the electrical storage cell is a rechargeable electrical storage cell.
- 63. (Original) An anode of an electrical storage cell comprising the coated graphitic particles of claim 56.
- 64. (Original) An anode of an electrical storage cell according to claim 63, wherein the electrical storage cell is a rechargeable electrical storage cell.
- 65. (Original) A method for the manufacture of an electrical storage cell, wherein the method comprises incorporating into an anode of the electrical storage cell coated graphitic materials comprising coated carbonaceous particles having a coating layer formed of an oxidized, carbon residue forming material.

- 66. (Original) The process of claim 2, wherein the coating of the particles is enhanced by effecting a partial precipitation of the carbon residue forming material onto the surface of the carbonaceous particles.
- 67. (Original) The process of claim 66, wherein the partial precipitation is effected by dilution of a concentrated solution of a carbon residue forming material by adding more of the same or one or more different solvents.
- 68. (Original) The process of claim 67, wherein the ratio of solvent to carbon residue forming material in the concentrated solution is 2:1 or less and the ratio of solvent to carbon residue forming material in the diluted solution is greater than 2:1.
- 69. (Original) The process of claim 67, wherein the ratio of solvent to carbon residue forming material in the concentrated solution is 2:1 or less and the ratio of solvent to carbon residue forming material in the diluted solution is greater than 5:1.

- 70. (Original) The process of claim 66, wherein partial precipitation of the carbon residue forming material is effected by cooling the mixture of carbonaceous particles and carbon residue forming material during the coating step.
- 71. (Original) The process of claim 2, wherein the coating of carbon residue forming material is deposited under ambient or higher pressure.
- 72. (Original) The coated carbonaceous material of claim 55 which, when used as an anode material in a lithium ion battery, shows a first cycle charge efficiency greater than 90% at a cut-off potential of 0.5 volts versus lithium metal.

REMARKS

The above amendatory action taken in the claims is solely for the purpose of avoiding claim fees that would otherwise accrue due to the presence of multiple dependent claims.

Respectfully submirred

LADAS & PARRY 26 West 61st. Street

New York, New York 10023

Reg. 31053

Tel. (212) 708-1915

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Practitioner's Docket No. <u>U015296-2</u>

June 21, 2005

Zhenhua Mao U.S. Serial No. 10/501,311

International Appln. No.: PCT/US03/02972 International Filing Date: January 31, 2003

Completion of Filing Requirements for International Application; Declaration; Copy of Notice of Missing Requirements; Check \$130.00; Preliminary Amendment

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